

PATENT SPECIFICATION

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(54) DEWATERING OF SEWAGE SLUDGES

(71) We, ALLIED COLLOIDS LIMITED, a British Company, of P.O. Box 38, Low Moor, Bradford, West Yorkshire, BD12 0JZ, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

5 The problem of solids disposal from water pollution control plants has received much attention in recent years and many mechanical dewatering devices are now in operation which remove water from sewage sludges enabling the solid residues to be more readily and economically disposed of, for example by incineration.

10 The performance of mechanical dewatering devices depends on a number of factors but perhaps the most important of these is the effective pre-treatment of the feed slurry by flocculants or coagulants. Such chemicals improve the filtration characteristics of the feed material by flocculating the small solid particles allowing the liquid phase to more easily separate from the solid phase.

15 It is known that cationic polymeric flocculants are generally more effective as dewatering aids for organic sludges than anionic or non-ionic polymer flocculants. It is also known that these cationic polymeric flocculants or polyelectrolytes offer many advantages over the inorganic coagulants which have traditionally been used. For example one important advantage is that the organic polyelectrolytes do not produce corrosive combustion products on incineration.

20 Typical amongst the many disclosures of cationic flocculants for use in flocculating sewage and other organic suspensions are U.S. Patent Specifications Nos. 3,171,805, 3,409,546 and 3,414,514 and British Patent Specification No. 1,373,034.

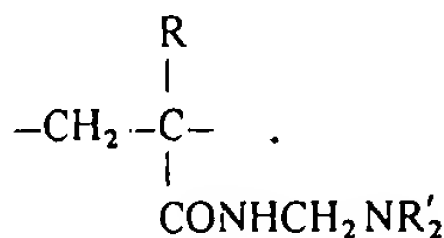
25 There are various ways of separating the flocculated solids from the supernatant liquid. In the simplest form separation is achieved merely by sedimentation or by drainage under ambient pressure through a filter. However common mechanical dewatering devices include centrifuges, vacuum filters and pressure filtration devices.

30 Although many of the known flocculation and separation processes described give very good dewatering, this being due to a large part to the success of the flocculant used, it is a fact that pressure filtration devices have not been used with modern polyelectrolyte flocculants as successfully and as widely as might be expected for the dewatering of organic suspensions such as sewage sludges apparently because all attempts to use known cationic polymeric flocculants for flocculation prior to pressure filtration have given rather poor results. Additions of materials such as the product of Example 1 of British Specification No. 1,373,034 certainly gives some improvement in dewatering by pressure filtration but the improvement is not as great as is desirable for commercial purposes. Indeed some polymers that might be expected to be good flocculants worsen rather than improve high pressure filtration characteristics. Thus some cationic polyacrylamides have been found to worsen the pressure filtration characteristics of sewage sludges.

40 Disclosures such as U.S. Patent Specification No. 3,171,805 suggest that various polymers of a wide range of molecular weights can be used in sewage liquid-solids separation by settling, optionally combined with filtration, but there is no suggestion that any of the range of polymers disclosed would be useful in a pressure filtration process and commercially there is a great need for a polymeric flocculant that will be very successful in pressure filtration.

45 We have now surprisingly found that good results are obtained in pressure filtration if there is used as flocculant a polymer having a particularly high molecular weight and a particularly high content of aminomethylated methacrylamide or acrylamide groups.

Thus a process according to the invention for dewatering an organic suspension comprises flocculating the suspension by addition of a polymer and subjecting the flocculated product to pressure filtration, in which process the polymer contains at least 60 mole per cent groups of the formula



or a salt or quaternary ammonium salt thereof and that has a viscosity as measured on a 5% solution containing 3% sodium chloride by a Brookfield viscometer at 25°C (10 rpm Spindle No. 5 or 6) of at least 4,600 cps. The viscosity specified is equivalent to a molecular weight of about or slightly above 3×10^6 , i.e. around 3.2×10^6 .

In the formula given above R represents hydrogen or methyl and the radicals R', which may be the same or different, are lower alkyl or hydroxy lower alkyl or, together with the nitrogen atom to which they are attached, they may form a cyclic group of 5 to 7 carbon atoms. Lower alkyl groups contain 4 carbon atoms or less and preferred groups are methyl and ethyl. R is preferably hydrogen. The polymer can be in the free base form or in the form of an acid salt or it may be in the form of a quaternary salt. The quaternising radical can be, for instance, lower alkyl, preferably methyl. The anion of the salt is preferably methyl sulphate, ethyl sulphate or chloride.

The polymer preferably contains at least 70% and most preferably at least 80% of the specified units. Preferably it has a viscosity, specified as defined above, of at least 12,000 and most preferably of at least 16,000 cps with best results being obtained with viscosities above 20,000 or 22,000 cps. Thus in general the molecular weight of the product is above $3.5-4 \times 10^6$ and most preferably is above 4.5×10^6 .

In the polymers some or all of any recurring units other than the specified units may be acrylamide or methacrylamide units. The polymer may contain also other units derived from other vinyl monomers. These may be introduced primarily for the purpose of reducing the cost of the product. Such units may be derived from various water soluble monomers, such as acrylic acid or methacrylic acid or from water insoluble monomers such as vinyl acetate, methyl acrylate, acrylonitrile or styrene. The inclusion of some of these other units may result in a reduction in the water solubility of the polymer and naturally care must be taken to ensure that the nature and amount of such comonomers is such that the polymer remains sufficiently water soluble for the intended purpose.

The polymer is preferably prepared by forming a polymer containing acrylamide or methacrylamide units and then aminomethylating and optionally quaternising sufficient of these to give the required product.

The amide groups may be aminomethylated by reacting the homopolymer with formaldehyde and a suitable secondary amine. Preferred secondary amines are dimethylamine and diethylamine but others that may be used include for example piperidine and di-(β-hydroxy ethyl) amine. The amounts of formaldehyde and amine used for the aminomethylation are selected to give the desired degree of aminomethylation. The tertiary aminomethyl groups may then be quaternised if desired, for example by causing the polymer to react with a suitable alkylation agent, for example dimethyl sulphate, methyl chloride, iodide or bromide, or corresponding compounds in which the methyl radical is replaced by another alkyl radical, for instance ethyl.

The organic suspension dewatered in the invention may be derived from raw and/or processed sewage or may be obtained from a sedimentation stage to remove some of the liquid therefrom. The solids may have been subjected to a biological anaerobic digestion stage to produce a digested sludge. Usually the sludge has a solids content of from about 1% to about 8% by weight although under certain circumstances it can have a higher or lower solid content.

Dewatering can be effected in the invention by adding an appropriate amount of one of the specified polymers to the sludge prior to physical removal of the water. The addition is usually effected by adding an aqueous solution of the polymer for example having a concentration of from 0.01 to 1% and preferably from 0.05 to 0.2%. The amount of polymer used may vary according to the suspension being treated and according to the degree of dewatering required. Typical addition rates for a sewage sludge are in the range 0.1 to 0.5% of polymer based on the total weight of sludge solids. Addition may be by conventional methods and agitation of the mixture of sewage and polyelectrolyte is necessary to bring about flocculation.

Pressure filtration may then be carried out on the flocculated product in conventional manner such as by use of a filter press, a compression filter or a filter belt press. In general the pressure during filtration will always be around 50 kilopascals (kPa) or more, this being typical for filter belt presses. However the invention is of most value at higher pressures, such as are obtained in filter presses. Thus the invention is preferably carried out in combination with filtration on a filter press producing a maximum pressure of between 400 and 700 kPa or with a compression filter

producing a maximum pressure of above 400 kPa and often above 1000 kPa.

The invention is illustrated by the following Examples.

Example 1

A range of polyacrylamides of varying molecular weight was prepared at 12.5% solids and their 1% solution viscosities determined using the suspended level technique. These polymers were then diluted to 3.0% solids, sodium chloride was added to 3.0% and the polymers were aminomethylated with one equivalent of formaldehyde and 1.1 equivalents of dimethylamine. After five days at room temperature the reaction came to an equilibrium with 85% of the amide groups aminomethylated. The Brookfield viscosities of the 5% active products were then determined. The results are given in Table 1.

Table 1

| Aminomethylated Polymer | 1% solution viscosity of the Polyacrylamide (cs) | Molecular weight of polyacrylamide | Aminomethylated product viscosity (5% active) (cps) | Molecular weight of aminomethylated |
|-------------------------|--|------------------------------------|---|-------------------------------------|
| P ₁ | 7.95 | 10 ⁶ | 1,000 | 1.7 x 10 ⁶ |
| P ₂ | 8.95 | | 1,800 | |
| P ₃ | 11.5 | | 2,500 | |
| P ₄ | 20.2 | 1.9 x 10 ⁶ | 4,600 | 3.2 x 10 ⁶ |
| P ₅ | 30.0 | | 12,000 | |
| P ₆ | 40.9 | | 16,500 | |
| P ₇ | 50.0 | | 21,500 | |
| P ₈ | 58.0 | 2.8 x 10 ⁶ | 24,000 | 4.7 x 10 ⁶ |

In this table polymers P₄ to P₈ only are useable in the invention.

Example 2

Digested sewage sludges from two plants were subjected to standard laboratory techniques for measuring the Capillary Suction Time (CST) by adding to each a given amount of a polyelectrolyte under varying conditions of shear determined by the stirring time at 1000 rpm. The effect of the application of shear, and in particular the higher shear values given below, is a reliable indication of how the polymers will act in a pressure filter. The polymers used included P₁ to P₈ of Example 1 and the following:—

Product A—a commercially available high molecular weight quaternised aminomethylated polyacrylamide as described in British Patent Specification No. 1,373,034 and having a molecular weight of 10⁶.

Product B—a commercially available polyacrylamide with approximately 50% (molar) of aminomethylated acrylamide groups and having a molecular weight of 3 x 10⁶.

Product C—a commercially available high molecular weight cationic polyacrylamide containing 10% (molar) of dimethylaminoethylacrylate groups quaternised with dimethyl sulphate and having a molecular weight of 3.5 x 10⁶.

The CST values given in Table 2 were measured at 175 g. of polyelectrolyte per m³ of the first sludge and at 75 g. of polyelectrolyte per m³ of the second sludge.

Table 2

| Polyelectrolyte | CST for 1st sludge | | | CST for 2nd sludge | | |
|-----------------|--------------------|----|----|--------------------|-----|-----|
| | Shear (sec) 10 | 20 | 30 | Shear (sec) 5 | 15 | 30 |
| Product A | 24 | 33 | 54 | 197 | 317 | 520 |
| " B | 41 | 47 | 64 | 40 | 131 | 290 |
| " C | 20 | 22 | 46 | — | — | — |
| P ₁ | 16 | 32 | 37 | 99 | 203 | 320 |
| P ₂ | 10 | 23 | 32 | 77 | 140 | 269 |
| P ₃ | 10 | 21 | 27 | 43 | 115 | 171 |
| P ₄ | 8 | 17 | 28 | 20 | 83 | 159 |
| P ₅ | 9 | 18 | 24 | 22 | 41 | 81 |
| P ₆ | 6 | 11 | 18 | 22 | 23 | 50 |
| P ₇ | 10 | 13 | 15 | 18 | 21 | 48 |
| P ₈ | 8 | 14 | 22 | 24 | 22 | 44 |

From the figures given in this Table it can be seen that products P₄ to P₈ all have lower CST values, and are therefore better as flocculants, under the conditions specified, than the Products A, B, C and P₁ to P₃. Further it is seen that in general increasing molecular weight gives improved properties. Further comparison of P₄ with Product B suggests that increasing the proportion of aminomethylated amide groups improves the properties of the polymer.

Example 3

In order to determine the effect of increasing the amount of conversion of the amide groups to aminomethylated groups a sewage sludge was treated with an acrylamide polymer of the type P₅ but with varying amounts of dimethylaminomethylation and tested in a laboratory filter press. The results, given in Table 3, show that the dewaterability increases with increases in aminomethylation in the range 35 to 85%.

Table 3

| | % aminomethylation | % cake solids |
|------|--------------------|---------------|
| 35.5 | 23.3 | |
| 49.0 | 24.6 | |
| 68.7 | 26.7 | |
| 77.5 | 27.3 | |
| 85.8 | 27.6 | |

In this test cake solids values above about 25% are satisfactory.

Example 4

In order to obtain a more realistic assessment of the effectiveness of the polymers in plant conditions, tests were carried out on a laboratory plate and frame press using a mixed activated and secondary digested sewage sludge with a selected dose of 2.2% of an inorganic coagulant (aluminium chlorohydrate) on a dry solids basis and cost equivalent doses of polyelectrolytes. Each filtration was carried out for a period of time sufficient to product an asymptotic filtration rate at a pressure of 482 kPa. The results are given in table 4.

Table 4

| Chemical Additive | Cake Solids (%) | Pressing Time (hours) |
|-------------------------|-----------------|-----------------------|
| Aluminium chlorohydrate | 32.9 | 3.5 |
| Product A | 31.6 | 2.25 |
| P ₈ | 33.0 | 1.0 |

It can be seen that a large reduction of pressing time is made possible by the use of the aminomethylated polyacrylamide P₈.

Example 5

A primary sludge in 300 ml aliquots was subjected to a gradual increase of up to 482 kPa in a laboratory piston press during a period of one hour. Chemical dosages for comparison purposes were based on 1.1% of aluminium chlorohydrate on a dry weight basis and a cost-equivalent dose of the polyelectrolytes. The results obtained are shown in table 5.

Table 5

| Additive | Cake Solids (%) | Cake Thickness (mm) |
|-------------------------|-----------------|---------------------|
| Aluminium chlorohydrate | 38.8 | 9 |
| P ₈ | 43.5 | 7 |
| Product C | 30.4 | 10.5 |

The lower cake thickness and higher % cake solids shows the advantage of using P₈.

Example 6

CST measurements were made for a sewage sludge from a secondary digester by conditioning it with 200 g. of Product A or P₈, per m³ of sludge, at three levels of shear determined by the stirring time at 1000 rpm. The results are given in Table 6.

Table 6

| Polyelectrolyte | CST | | |
|-----------------|-------------|----|-----|
| | Shear (sec) | | |
| | 5 | 15 | 30 |
| Product A | 20 | 51 | 123 |
| P ₈ | 26 | 27 | 47 |

Example 7

The polyelectrolytes of Example 6 were compared using a piston press and the sludge of Example 6.

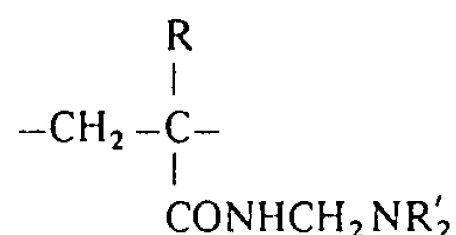
300 ml aliquots of the treated sludge were subjected to a gradual pressure increase of up to 482 kPa during a period of 1 hour. The comparison was carried out using a dose of 250 g/m³ in each case. The results are given in Table 7.

Table 7

| Polyelectrolyte | Cake thickness (mm) | Cake solids (%) |
|-----------------|---------------------|-----------------|
| Product A | 35 | 12.1 |
| P ₈ | 23 | 17.2 |

WHAT WE CLAIM IS:—

1. A process for dewatering an organic suspension comprising flocculating the suspension by addition of a polymer and subjecting the flocculated product to pressure filtration, in which process the polymer contains at least 60 mole per cent groups of the formula



or a salt or quaternary ammonium salt thereof wherein R represents hydrogen or methyl and the radicals R', which may be the same or different, are alkyl or hydroxyalkyl groups containing up to 4 carbon atoms or, together with the nitrogen atom to which they are attached, they form a cyclic group of 5 to 7 carbon atoms, and the polymer has a viscosity as measured on a 5% solution containing 3% sodium chloride by a Brookfield viscometer at 20°C (10 r.p.m. spindle no. 5 or 6) of at least 4,600 c.p.s.

2. A process according to claim 1 in which R represents hydrogen and the radicals R', which may be the same or different, are methyl or ethyl.

3. A process according to claim 1 or claim 2 in which the viscosity is at least 12,000 c.p.s.
4. A process according to claim 3 in which the viscosity is at least 20,000 c.p.s.
5. A process according to any preceding claim in which the pressure filtration is conducted on a filter press or compression filter at a maximum pressure of above 400 kPa.
- 5 6. A process according to any preceding claim in which the organic suspension is a sewage sludge. 5
7. A process according to claim 1 substantially as herein described with reference to any of Examples 2 to 6.

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